

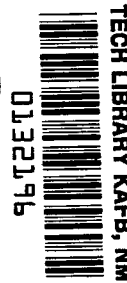
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HOT-SALT-STRESS-CORROSION  
CRACKING AND ITS EFFECT ON TENSILE  
AND STRESS-RUPTURE PROPERTIES OF  
Ti-6Al-4V TITANIUM-ALLOY SHEET

*by Dick M. Royster*

*Langley Research Center*

*Langley Station, Hampton, Va.*



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16. Abstract  Self-stressed, tensile, and stress-rupture specimens were made from 0.040-inch-thick (1.0 mm) annealed sheet. The self-stressed specimens, with stresses of 25 and 50 ksi (170 and 340 MN/m <sup>2</sup> ), were sprayed with a 3.4-percent sodium chloride solution and exposed at temperatures from 500° F to 600° F (530 K to 590 K) up to 10 000 hours. Cracking was obtained after approximately 20 hours at 600° F for the 50 ksi stress, but no cracking was noted at 500° F for up to 10 000 hours at either stress. The tensile specimens were salt coated in the same manner as the self-stressed specimens and exposed up to 1600 hours at temperatures from 550° F to 650° F (560 K to 620 K) and 50 ksi stress to develop and grow hot-salt-stress-corrosion cracks. When tested at room temperature, the presence of stress-corrosion cracks resulted in a large reduction in elongation. There was also a large decrease in tensile strength for the specimens exposed at 650° F. The salt-coated stress-rupture specimens were exposed at temperatures from 600° F to 700° F (590 K to 650 K) for stresses of 50 to 100 ksi (340 to 690 MN/m <sup>2</sup> ) until rupture occurred. A time-temperature parameter analysis was made of both the crack-initiation and stress-rupture data. A difference between the time for crack initiation and the time for stress rupture by a factor on the order of 300 was indicated.		13. Type of Report and Period Covered Technical Note	
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SUMMARY

An extensive experimental investigation of hot-salt-stress-corrosion cracking on 0.040-inch-thick (1.0 mm) Ti-6Al-4V titanium-alloy (annealed) sheet was conducted with self-stressed, tensile, and stress-rupture specimens. The self-stressed specimens, with stresses of 25 and 50 ksi (170 and 340 MN/m<sup>2</sup>), were sprayed with a 3.4-percent sodium chloride (NaCl) solution and exposed at temperatures from 500° F to 600° F (530 K to 590 K) up to 10 000 hours. Cracking was obtained after approximately 20 hours at 600° F (590 K) for the 50 ksi (340 MN/m<sup>2</sup>) stress, but no cracking was noted at 500° F (530 K) for up to 10 000 hours at either stress level. The threshold stresses for onset of hot-salt-stress-corrosion cracking decreased with an increase in exposure temperature and time.

The tensile specimens were salt coated in the same manner as the self-stressed specimens and exposed up to 1600 hours at temperatures from 550° F to 650° F (560 K to 620 K) and 50 ksi (340 MN/m<sup>2</sup>) stress to develop and grow hot-salt-stress-corrosion cracks. When tested at room temperature, the presence of stress-corrosion cracks resulted in a large reduction in elongation. There was also a large decrease in tensile strength for the specimens exposed at 650° F (620 K). Maximum crack depths were found to increase with exposure time and were nearly the same for both longitudinal and transverse specimens.

The salt-coated stress-rupture specimens were exposed at temperatures from 600° F to 700° F (590 K to 650 K) for stresses of 50 to 100 ksi (340 to 690 MN/m<sup>2</sup>) until rupture occurred. A time-temperature parameter analysis was made of both the crack initiation and stress-rupture data. A difference between the time for crack initiation and the time for stress rupture by a factor on the order of 300 was indicated.

A comparison of the effect of hot-salt-stress-corrosion cracks on Ti-8Al-1Mo-1V and Ti-6Al-4V titanium alloys from tests conducted at the Langley Research Center revealed little significant difference in crack initiation times for the two alloys at 600° F and 550° F (590 K and 560 K). The cracks were deeper and more extensive in

the Ti-8Al-1Mo-1V titanium alloy than in the Ti-6Al-4V titanium alloy. Also, the effect of the cracks was more detrimental to the tensile strength of Ti-8Al-1Mo-1V than to that of Ti-6Al-4V alloy.

## INTRODUCTION

The susceptibility of various titanium-alloy sheet materials to hot-salt-stress corrosion has been considered a possible detriment to their use as skin materials for a supersonic transport operating at speeds up to Mach 3 (ref. 1). An initial program at the Langley Research Center to study the susceptibility of titanium alloys to hot-salt-stress corrosion (ref. 2) indicated that several of the alloys were susceptible to this type of corrosion. Consequently, an extensive investigation of hot-salt-stress corrosion of titanium-alloy sheet was undertaken. Ti-8Al-1Mo-1V (duplex annealed) and Ti-6Al-4V (annealed) alloys were emphasized because of their desirable mechanical properties. Test results on the Ti-8Al-1Mo-1V alloy have been presented in references 3, 4, and 5.

The present paper summarizes the results of the investigation of hot-salt-stress-corrosion cracking of Ti-6Al-4V titanium-alloy sheet at temperatures from 500° F to 700° F (530 K to 650 K) for exposures up to 10 000 hours at stresses from 25 to 100 ksi (170 to 690 MN/m<sup>2</sup>). Data are shown on the exposures times, stresses, and temperatures at which hot-salt-stress-corrosion cracking was initiated. Also included are the extent and depth of cracks and their subsequent effect on the room-temperature residual tensile properties after exposure at 550° F to 650° F (560 K to 620 K) for up to 1600 hours at 50 ksi (340 MN/m<sup>2</sup>) stress. Time to rupture for salt-coated stress-rupture specimens was also determined at temperatures from 600° F to 700° F (590 K to 650 K) and stresses from 50 to 100 ksi (340 to 690 MN/m<sup>2</sup>). A time-temperature parameter analysis was made of both the crack-initiation and stress-rupture data and an approximate relationship between times for crack initiation and stress rupture was indicated.

The units used for the physical quantities defined in the paper are given both in U.S. Customary Units and in the International System of Units (SI) (ref. 6). Factors relating the two systems are given in the appendix. For the data in the tables and figures, the SI values are more exact than those given in the text.

## SPECIMENS AND EXPERIMENTAL PROCEDURES

### Material

All specimens were fabricated from three sheets of Ti-6Al-4V titanium alloy (0.040 inch (1.0 mm) nominal thickness) supplied from the same heat in the annealed condition. Annealing consisted of 1 hour at 1475° F (1075 K) with a furnace cool to

1300<sup>0</sup> F (980 K) and air cooling to room temperature. The following information on the chemical composition of this material was supplied by the producer:

Element	Weight, percent
Al	5.9
V	4.1
O <sub>2</sub>	.11
Fe	.05
C	.025
N	.015
H <sub>2</sub>	.008
Ti	Balance

### Specimens

Self-stressed, tensile, and stress-rupture specimens (fig. 1) were utilized in this investigation. Some specimens were given a salt coating and exposed in ovens. Other specimens that had similar exposures in the ovens without a salt coating are herein defined as control specimens. Uncoated, unexposed refers to specimens tested in the as-received condition without salt or elevated-temperature exposure.

Self-stressed specimens.- In fabricating the self-stressed specimens (fig. 1(a)), flat strips were sheared from the titanium-alloy sheet so that the longitudinal axis was parallel to the rolling direction. The strips were then hand deburred, machined to desired dimensions, and deburred a second time.

In general, the self-stressed specimen is constructed by bending up the ends of each 1/4- by 4-inch (0.60 by 10.20 cm) strip to some predetermined bend angle and spot-welding the ends of two strips together to induce a uniform curvature. Bending stresses corresponding to this curvature are determined from geometrical and stress-strain relationships (ref. 7) by using the measured value  $d$ , the distance between the strips at the center of the specimen. (See fig. 1(a).) Stresses are increased by increasing the bend angle of the strip; thus  $d$  is increased. The self-stressed specimens were designed for room-temperature maximum outer-fiber tensile stresses of 50 and 25 ksi (340 and 170 MN/m<sup>2</sup>). The table in figure 1 shows the calculated tensile stresses for each value of  $d$ . Specific details of the fabrication and cleaning processes are given in reference 2.

Tensile and stress-rupture specimens.- For the tensile specimens, blanks were sheared from the titanium-alloy sheet in both the longitudinal and transverse directions of rolling, and the specimens were machined into the desired configuration (fig. 1(b)). The

tensile specimen conforms to the specification prescribed by the American Society for Testing and Materials (ref. 8). For the stress-rupture specimens, blanks were sheared from the titanium-alloy sheet in only the longitudinal direction of rolling. The specimens were then machined into the desired configuration (fig. 1(c)).

### Salt Coating

Self-stressed specimens.- Salt coatings were applied to the self-stressed specimens by spraying on a 3.4 percent by weight solution of sodium chloride and distilled water. Usually 6 or 7 spray applications, each followed by an air-drying cycle in an oven at 200° F (370 K), were required before a thin film of salt of approximately 10.0 mg/in<sup>2</sup> (16 g/m<sup>2</sup>) would accumulate on one of the two convex outer surfaces of each specimen. The specimen was then turned over and the remaining outer surface was salt coated in the same manner. In actual flight, salt deposits of up to 1 mg/in<sup>2</sup> (1.6 g/m<sup>2</sup>) can result on engine components after exposure to severe environmental conditions (helicopter hovering over the ocean) and local concentrations up to 10 times the average have been measured (ref. 9).

Tensile and stress-rupture specimens.- Salt coatings were applied to the tensile and stress-rupture specimens by suspending them horizontally in an oven, with a flat surface up, and spraying the salt solution over the entire reduced section of each specimen. The same spraying technique and drying procedure used for the self-stressed specimens were utilized in salt coating both flat surfaces of these specimens.

### Elevated Temperature Exposure

Self-stressed specimens.- Salt-coated and control specimens were subjected to continuous heating at 500° F, 550° F, or 600° F (530 K, 560 K, or 590 K) at sea-level atmosphere in circulating air ovens for various exposures up to 10 000 hours. Specimens were removed from the ovens after selected exposure times and were mechanically tested at room temperature.

Tensile specimens.- The salt-coated tensile specimens were exposed at 550° F, 600° F, or 650° F (560 K, 590 K, or 620 K) at a stress of 50 ksi (340 MN/m<sup>2</sup>) for selected exposure times up to 1600 hours in conventional creep testing machines (dead-weight loading machines) equipped with tube furnaces in order to develop and grow hot-salt-stress-corrosion cracks. After selected exposures, the specimens were removed from the furnaces and creep machines and tested in tension at room temperature. Control specimens were also exposed at 600° F (590 K) for 1600 hours at a stress of 50 ksi (340 MN/m<sup>2</sup>) to determine the effect of the stress and elevated-temperature exposure on the residual tensile properties.

Stress-rupture specimens.- The salt-coated stress-rupture specimens were exposed at 600° F, 650° F, or 700° F (590 K, 620 K, or 650 K) at stresses of 50 to 100 ksi (340 to 690 MN/m<sup>2</sup>) in the same creep testing machines and tube furnaces that were used for the tensile specimens. Each specimen was exposed continuously to the selected stress and temperature until rupture occurred. Control specimens were exposed at 650° F and 700° F (620 K and 650 K) at stresses of 90 and 80 ksi (620 and 550 MN/m<sup>2</sup>), respectively, to determine the stress-rupture strength of the uncoated material.

### Mechanical Tests

Self-stressed specimens.- The effects of the hot-salt-stress corrosion on the self-stressed specimens were determined by a specimen shortening test at room temperature. (See fig. 2.) In these tests the specimens were loaded in compression in a 120-kip-capacity (530 kN) hydraulic testing machine. A special fixture (ref. 10) was used to align upper and lower clamps with the specimen. The clamps supported the specimen vertically in the testing machine during the compression test. The load was applied at a constant head speed of 0.1 in./min (40  $\mu$ m/s) until the maximum load was reached. Then an increased head speed of 0.4 in./min (170  $\mu$ m/s) was applied until fracture occurred at which time loading was stopped. The head displacement, which is equivalent to specimen shortening, was then measured with a 6-inch (15 cm) scale having 0.01-inch (0.25 mm) graduations.

A more detailed discussion of this test is found in reference 3. In general, the results obtained can be explained by the following discussion. Uncoated, unexposed specimens develop the maximum shortening  $\delta_0$ . If stress-corrosion cracking occurs in the salt-coated specimens, fracture will occur at a reduced amount of shortening. The change in shortening of the control specimens after the different oven exposures is a measure of the effect of the exposures on the bend ductility of the material. The ratio  $\delta/\delta_0$  - the shortening of the exposed specimens to that of the uncoated, unexposed specimens - is a measure of the reduction in bend ductility due to hot-salt-stress-corrosion cracking or exposure embrittlement of the material or both and is defined as relative shortening.

Tensile specimens.- The residual strength of hot-salt-stress-corrosion cracked tensile specimens of Ti-6Al-4V alloy was determined by room-temperature tensile tests after the elevated-temperature stress exposure. These tests were conducted under controlled strain-rate conditions in a hydraulic testing machine of 100-kip (445 kN) capacity and equipped with a load-strain recorder. A strain rate of 0.005 per minute was maintained through the yield stress at which point the rate was increased to 0.050 per minute until fracture occurred. Wire strain gages mounted back to back on the specimens were

used to measure strain in the room-temperature tests. The uniform elongation over 1 inch (2.54 cm) was measured from the increase in length between transverse parallel lines spaced 0.25 inch (0.60 cm) apart on the reduced section of the specimen in the region where necking did not occur. The elongation measurements in 2 inches (5 cm) included the neck-down and fracture region of the test specimen. The measurements were made with a 6-inch (15 cm) scale having 0.01-inch (0.25 mm) graduations.

Stress-rupture specimens.- The stress-rupture properties of salt-coated stress-rupture specimens were determined by continuous exposure of the specimens to the stress and temperature until rupture occurred. Exposure times to rupture were obtained from digital elapsed-test-time indicators that cut off automatically when the specimens ruptured.

### Crack Detection and Measurements

Self-stressed specimens were examined after elevated-temperature exposure and room-temperature compression tests for the presence of hot-salt-stress-corrosion cracks. An etch that has been used in chemical milling of titanium alloys was found to be very effective in revealing cracks (ref. 11). The etch consists of 1 part concentrated hydrofluoric acid (HF), 6 parts hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) (30 percent concentration), and 3 parts water, by volume. The specimen to be etched is dipped in the solution at room temperature for 30 seconds, then removed and washed in distilled water. If cracks are present, they are revealed without additional surface preparation and can be readily seen at low magnification or with the naked eye.

In order to determine the details of the depth and metallurgical characteristics of the cracks in the tensile specimens, the failed specimens were edge mounted in plastic and wet ground to remove about 1/8 inch (0.3 cm) of material from the edge. The specimens were polished and then chemically etched at room temperature for 15 seconds with a solution of 97 percent water, 2 percent nitric acid ( $\text{HNO}_3$ ), and 1 percent hydrofluoric acid (HF), by volume. Specimens were also mounted face up to study the extent of surface cracking.

An examination of the fractured surface of tensile and stress-rupture specimens revealed small discolored areas around the perimeter of the fracture which were identified as hot-salt-stress-corrosion cracks from which failures of the specimens were initiated. The discoloration occurred as a result of the elevated-temperature exposure oxidizing the material in the crack. The depth of the discoloration (crack) was measured on the tensile specimens by a light microscope with a filar micrometer eyepiece.



## RESULTS AND DISCUSSION

### Hot-Salt-Stress-Corrosion Crack Initiation

The results of the hot-salt-stress-corrosion tests of Ti-6Al-4V titanium-alloy sheet stressed at 50 and 25 ksi (340 and 170 MN/m<sup>2</sup>) and exposed up to 10 000 hours at 500° F to 600° F (530 K to 590 K) are shown in figure 3. Although the data show considerable variability, the increasing severity of cracking with exposure time is evidenced by a reduction in the relative shortening  $\delta/\delta_0$ . Also shown in figure 3 is the approximate threshold time for hot-salt-stress-corrosion cracking for the test stresses and temperatures. The thresholds were selected after consideration of both the relative shortening curves in figure 3 and a microscopic examination of the test specimens. This threshold is defined as the approximate time when the relative shortening curve for the salt-coated specimens begins to deviate significantly from the relative shortening curve for the control specimens. It should be noted that because of the nature of the results, crack initiation times cannot be determined precisely. The existence of cracks in the specimens for exposures to the right of the threshold of the curves was verified by chemical etch and microscopic examination. Specimens exposed for combinations of temperature, time, and stress to the left of the threshold showed no cracks after exposure, mechanical testing, and examination.

The Ti-6Al-4V titanium-alloy control specimens appeared to be relatively unaffected by the different exposure conditions as indicated in figure 3 by the small reduction in relative shortening of the control specimens. This reduction (no more than 4 percent) may be due to normal scatter in the test. Microscopic examination of the control specimens after exposure showed no evidence of cracking.

The most severe cracking occurred at the highest temperature (600° F (590 K)) and stress (50 ksi (340 MN/m<sup>2</sup>)). (See fig. 3(a).) The results indicate that cracking may be expected after approximately 20 hours exposure at 600° F (590 K) for a 50 ksi (340 MN/m<sup>2</sup>) stress and after approximately 230 hours exposure at the same temperature for a 25 ksi (170 MN/m<sup>2</sup>) stress. Cracking was initiated at 550° F (560 K) in approximately 110 hours for the specimens at a 50 ksi (340 MN/m<sup>2</sup>) stress and approximately 2200 hours for the specimens at a 25 ksi (170 MN/m<sup>2</sup>) stress (fig. 3(b)). No evidence of cracking was found at 500° F (530 K) for the two stresses. (See fig. 3(c).) The severity of the cracking, as measured by relative shortening, decreased with a decrease in stress and temperature.

Threshold curves for the initiation of hot-salt-stress-corrosion cracking for Ti-6Al-4V titanium-alloy sheet at 550° F and 600° F (560 K and 590 K) are shown in figure 4. The continuous curves were constructed from the thresholds indicated in figure 3. The faired curves give the stresses up to 50 ksi (340 MN/m<sup>2</sup>) and exposure times up to

10 000 hours at the test temperatures. To the left of any particular curve, no cracking occurred; to the right, cracking can be expected. No threshold curve is shown for 500° F (530 K) because no cracking was found at that temperature for the stresses and exposure times investigated.

The threshold curves for initiation of hot-salt-stress-corrosion cracking of Ti-8Al-1Mo-1V titanium alloy taken from reference 5 are shown as dashed curves in figure 4. There does not seem to be a significant difference between the results for crack initiation for these two alloys at 600° F and 550° F (590 K and 560 K) in these investigations except at 600° F (590 K) and 25 ksi (170 MN/m<sup>2</sup>), where the Ti-6Al-4V alloy had a considerably lower crack initiation time than the Ti-8Al-1Mo-1V alloy.

Although the threshold data in figure 4 for hot-salt-stress-corrosion cracking of Ti-6Al-4V are limited, the systematic trend in the data suggests that a time-temperature parameter may be applicable. Accordingly, the Orr-Sherby-Dorn parameter (ref. 12) was used to correlate the hot-salt-stress-corrosion crack-initiation data since it was found in reference 5 that this parameter could be used in predicting combinations of times and temperatures for crack initiation of Ti-8Al-1Mo-1V titanium alloy. In figure 5 the stress for crack initiation for Ti-6Al-4V titanium alloy is plotted against the logarithmic form of the Orr-Sherby-Dorn parameter  $\log t - \frac{\Delta H}{2.3RT_K}$  where  $t$  is the time in hours,  $\Delta H$  is activation energy (calculated to be 47 000 cal/mole (197.0 kJ/mole) as described in ref. 12),  $R$  is the gas constant (2.0 cal/mole-°K (8.4 J/mole-K)), and  $T_K$  is the temperature in Kelvin. The close grouping of the data points indicates good agreement over the temperatures and stresses investigated. A straight line or master curve averages these data points. Even though the data are limited, it appears that hot-salt-stress-corrosion crack initiation for Ti-6Al-4V can also be predicted with the Orr-Sherby-Dorn time-temperature parameter.

#### Effect of Stress-Corrosion Cracking on Tensile Properties

The results of the room-temperature tensile tests of the exposed, salt-coated tensile specimens as well as for the uncoated, unexposed specimens and control specimens are given in table I. Figure 6 shows the effect of the hot-salt-stress-corrosion cracks on residual tensile and yield strengths and uniform elongation at room temperature after exposures up to 1600 hours at 50 ksi (340 MN/m<sup>2</sup>) stress and temperatures from 550° F to 650° F (560 K to 620 K). The data for the uncoated, unexposed specimens and the control specimens are also shown in figure 6. At 600° F (590 K) the tensile strength of the cracked specimens was reduced in both the longitudinal and transverse directions (figs. 6(a) and (b)) with a maximum reduction of about 12 and 17 percent, respectively, after 1600 hours exposure. This reduction in strength is based on the strength of the uncoated, unexposed specimens. For the same exposure the control specimens showed

no reduction in tensile strength, which indicates that this combination of temperature and time had no effect on the tensile properties of the material.

For comparative purposes, data from reference 5 showed that the tensile strength of salt-coated Ti-8Al-1Mo-1V titanium alloy after 800 hours exposure at 50 ksi ( $340 \text{ MN/m}^2$ ) and  $600^\circ \text{ F}$  ( $590 \text{ K}$ ) was reduced in both the longitudinal and transverse directions by 12 and 22 percent, respectively. This reduction indicates that hot-salt-stress-corrosion cracking is more detrimental to the tensile strength of Ti-8Al-1Mo-1V titanium alloy than to the tensile strength of Ti-6Al-4V titanium alloy.

At  $650^\circ \text{ F}$  ( $620 \text{ K}$ ) after 400 hours exposure there was a reduction in tensile strength in both the longitudinal and transverse directions of about 49 and 46 percent, respectively. (See fig. 6.) The tensile strength of the specimens at  $550^\circ \text{ F}$  ( $560 \text{ K}$ ) showed little effect of the exposure except for the longitudinal specimens after 1200 and 1600 hours. In this region there is some scatter in both the  $550^\circ \text{ F}$  and  $600^\circ \text{ F}$  ( $560 \text{ K}$  and  $590 \text{ K}$ ) test data. Figure 6 indicates little effect on the yield strength of the corrosion cracked specimens at  $550^\circ \text{ F}$  ( $560 \text{ K}$ ). At  $600^\circ \text{ F}$  and  $650^\circ \text{ F}$  ( $590 \text{ K}$  and  $620 \text{ K}$ ) the yield strength data are limited as some specimens (both longitudinal and transverse) fractured before yield stress was reached. (See table I.)

This discussion points up the fact that a more serious problem than the decrease in tensile strength due to hot-salt-stress-corrosion cracking is the large reduction in uniform elongation obtained on the salt-coated specimens for all exposures. (See fig. 6.) On the basis of the loss in tensile strength and large reduction in elongation at  $550^\circ \text{ F}$  ( $560 \text{ K}$ ) and above, hot-salt-stress-corrosion cracking can have very detrimental effects on residual tensile properties.

It has been suggested from tensile data in reference 9 that hydrogen embrittlement was the cause of hot-salt, stress corrosion of titanium alloys. Those tests were conducted on Ti-8Al-1Mo-1V titanium alloy, 1-inch-thick (2.54 cm) bar stock in the mill annealed condition. In attempting to compare the elongation data of reference 9 with elongation data of this report and reference 5, it appears that the exposures had a more detrimental effect on elongation in the latter two investigations. However, different test conditions and a difference in material (bar stock in ref. 9 and sheet material in this report and ref. 5) could account for the nonagreement in test data.

### Crack Characteristics

Typical crack penetration in the Ti-6Al-4V titanium alloy is shown in figure 7(a) for an edge-mounted longitudinal tensile specimen which had been exposed to a 50 ksi ( $340 \text{ MN/m}^2$ ) stress at  $600^\circ \text{ F}$  ( $590 \text{ K}$ ) for 800 hours and tested at room temperature. An enlargement of one of the cracks (fig. 7(b)) shows that the corrosion attack is intergranular.

The extent of the cracking on tensile specimens can be seen in the photographs of figure 8. The flat surfaces shown are portions of the tensile specimens adjacent to the fracture. These specimens were tested at room temperature after they had been salt coated and exposed for 1600 hours at 600° F (590 K) at 50 ksi (340 MN/m<sup>2</sup>) stress. The load was applied horizontally in relation to the photographs in figure 8 and the cracks are normal to the applied load. The cracks are usually more visible and numerous near the edge of the fracture, which is on the left side of each photograph. The cracks are not dispersed uniformly across the specimen surface even though the entire surface was salt coated. Control specimens which had been loaded to failure in tension showed no evidence of cracking. On the basis of a comparison of photographs of cracks in test specimens with similar exposures, it appears that cracking of Ti-8Al-1Mo-1V titanium alloy (ref. 5) is more extensive than the cracking of Ti-6Al-4V titanium alloy.

A study of the fractured surface of the tensile specimens revealed small semicircular discolored areas located randomly around the perimeter of the fractured surface. (See fig. 9.) The discolored areas on the fracture surface are due to hot-salt-stress-corrosion cracks that were initiated during elevated temperature exposure at 50 ksi (340 MN/m<sup>2</sup>) stress. As the cracks opened, the wall of the cracks was oxidized, just as the surface of the specimens was oxidized. When the specimens were later tested to failure at room temperature, the appearance of the cracked discolored areas of the fracture surface was very pronounced when compared with the uncracked areas of the fracture surface.

Crack penetration measurements are correlated in figure 10 with exposure times up to 1600 hours at 50 ksi (340 MN/m<sup>2</sup>) stress for temperatures of 550° F, 600° F, and 650° F (560 K, 590 K, and 620 K). The curves give the maximum crack depths measured on the fractured surface for the tensile specimens in both the longitudinal and transverse directions. The crack penetrations are similar for both the longitudinal and transverse specimens for a given time and temperature. Maximum crack depths increase with exposure time. A comparison of the crack depths for Ti-8Al-1Mo-1V titanium alloy (ref. 5) and Ti-6Al-4V titanium alloy reveals that for the same exposure conditions the cracks are deeper in the Ti-8Al-1Mo-1V titanium alloy.

#### Effect of Stress-Corrosion Cracking on Stress-Rupture Properties

The results of the stress-rupture tests of salt-coated stress-rupture specimens exposed at 600° F, 650° F, or 700° F (590 K, 620 K, or 650 K) from stresses of 50 to 100 ksi (340 to 690 MN/m<sup>2</sup>) are shown in figure 11 and tabulated in table II. The curves are faired through the data points. Tests of control specimens exposed at 650° F and 700° F (620 K and 650 K) for 90 and 80 ksi (620 and 550 MN/m<sup>2</sup>) stresses were terminated after being exposed for 2500 hours without rupture.

A photograph of a typical ruptured surface of a salt-coated stress-ruptured specimen is shown in figure 12. The fracture looks similar to the fractured surface of the tensile specimens of figure 9 except that the picture-frame effect around the perimeter of the fracture produced by the cracks is more complete.

The stress-rupture data (fig. 11) are represented by a family of smooth curves decreasing in a consistent manner with temperature which suggest that a time-temperature parameter may be applicable. The Orr-Sherby-Dorn parameter  $\log t - \frac{\Delta H}{2.3RT_K}$  (ref. 12) was utilized and the correlation of the test results with a master curve for the three temperatures is shown in figure 13. The close grouping of the data points indicates good agreement over the temperatures and stresses investigated. It thus appears that the Orr-Sherby-Dorn time-temperature parameter can be used for predicting stress rupture of salt-coated stress-rupture specimens as well as for predicting crack initiation for this material.

#### Relation Between Crack Initiation and Stress Rupture

The relation between the times for stress rupture and crack initiation is shown in figure 14. The curves for crack initiation and stress rupture are obtained from the master curves in figures 5 and 13, respectively. The data points for crack initiation and stress rupture are shown for correlation with the predicted curves. The validity of the stress-rupture curves extrapolated at the low stresses may be questionable. On the basis of the limited data and calculated curves, there appears to be a difference between the time for crack initiation and the time for stress rupture by a factor on the order of 300.

#### SUMMARY OF RESULTS

The susceptibility of Ti-6Al-4V titanium-alloy sheet (annealed) to hot-salt-stress-corrosion cracking and the effect of cracks on residual tensile and stress-rupture properties are summarized for exposures up to 10 000 hours at temperatures from 500° F to 700° F (530 K to 650 K) with stresses from 25 to 100 ksi (170 to 690 MN/m<sup>2</sup>). The following observations are based upon the findings in this investigation:

1. Hot-salt-stress-corrosion cracking occurred after approximately 20 hours exposure at 600° F (590 K) and 50 ksi (340 MN/m<sup>2</sup>) stress. No cracking was found at 500° F (530 K) for exposures up to 10 000 hours. The threshold stresses decreased with an increase in exposure temperature and time. Crack initiation can be predicted over a range of stresses, times, and temperatures with the Orr-Sherby-Dorn time-temperature parameter.

2. The room-temperature tensile properties of the Ti-6Al-4V sheet were seriously affected by the development of hot-salt-stress-corrosion cracks, depending on exposure

temperature. At the 650<sup>o</sup> F (620 K) exposure temperature there was a large reduction (approximately 50 percent) in tensile strength after 400 hours exposure for both the longitudinal and transverse directions. At the 600<sup>o</sup> F (590 K) exposure temperature there was a reduction in strength after 1600 hours of 12 and 17 percent for the longitudinal and transverse directions, respectively. The room-temperature tensile elongation properties of the Ti-6Al-4V sheet were seriously affected by the development of cracks. Maximum crack depths increased with exposure time and were nearly the same for both the longitudinal and transverse directions.

3. The Orr-Sherby-Dorn time-temperature parameter can be used for predicting stress rupture of salt-coated stress-rupture specimens. A difference between the time for crack initiation and stress rupture by a factor on the order of 300 was indicated.

4. Where comparisons could be made between Ti-6Al-4V and Ti-8Al-1Mo-1V titanium alloys, it appears that in general there is little difference in crack initiation times for the two alloys over the test stresses at 600<sup>o</sup> F and 550<sup>o</sup> F (590 K and 560 K). Cracks are generally deeper and more extensive for the Ti-8Al-1Mo-1V titanium alloy than for the Ti-6Al-4V titanium alloy for given exposure conditions. For the same exposures, hot-salt-stress-corrosion cracking is more detrimental on the tensile strength of Ti-8Al-1Mo-1V titanium alloy than on the tensile strength of Ti-6Al-4V titanium alloy.

Langley Research Center,

National Aeronautics and Space Administration,

Langley Station, Hampton, Va., June 24, 1969.

## APPENDIX

### CONVERSION OF U.S. CUSTOMARY UNITS TO SI UNITS

The International System of Units (SI) was adopted by the Eleventh General Conference on Weights and Measures in Paris, October 1960. (See ref. 6.) Factors required for converting the U.S. Customary Units used herein to the International System of Units (SI) are given in the following table:

Physical quantity	U.S. Customary Unit	Conversion factor (*)	SI Unit
Force	lbf	4.44822	newtons (N)
Length	in.	0.0254	meters (m)
Stress	ksi	$6.895 \times 10^6$	newtons/meter <sup>2</sup> (N/m <sup>2</sup> )
Temperature	°F	$\frac{5}{9}(F + 459.67)$	Kelvin (K)
Energy	cal	4.184	joules (J)

\*Multiply value given in U.S. Customary Unit by conversion factor to obtain equivalent value in SI Unit.

Prefixes to indicate multiples of units are as follows:

Prefix	Multiple
giga (G)	$10^9$
mega (M)	$10^6$
kilo (k)	$10^3$
centi (c)	$10^{-2}$
milli (m)	$10^{-3}$
micro ( $\mu$ )	$10^{-6}$

## REFERENCES

1. Raring, Richard H.; Freeman, J. W.; Schultz, J. W.; and Voorhees, H. R.: Progress Report of the NASA Special Committee on Materials Research for Supersonic Transports. NASA TN D-1798, 1963.
2. Braski, David N.; and Heimerl, George J.: The Relative Susceptibility of Four Commercial Titanium Alloys to Salt Stress Corrosion at 550° F. NASA TN D-2011, 1963.
3. Heimerl, G. J.; Braski, D. N.; Royster, D. M.; and Dexter, H. B.: Salt Stress Corrosion of Ti-8Al-1Mo-1V Alloy Sheet at Elevated Temperatures. Stress-Corrosion Cracking of Titanium, Spec. Tech. Publ. No. 397, Amer. Soc. Testing Mater., c.1966, pp. 194-214.
4. Dexter, Howard B.: Salt Stress Corrosion of Residually Stressed Ti-8Al-1Mo-1V Alloy Sheet After Exposure at Elevated Temperatures. NASA TN D-3299, 1966.
5. Royster, Dick M.: Hot-Salt-Stress-Corrosion Cracking and Its Effect on Tensile Properties of Ti-8Al-1Mo-1V Titanium-Alloy Sheet. NASA TN D-4674, 1968.
6. Comm. on Metric Pract.: ASTM Metric Practice Guide. NBS Handbook 102, U.S. Dep. Com., Mar. 10, 1967.
7. Heimerl, J.; and Braski, D. N.: A Stress Corrosion Test for Structural Sheet Materials. Mater. Res. Stand., vol. 5, no. 1, Jan. 1965, pp. 18-22.
8. Anon.: Tentative Methods of Tension Testing of Metallic Materials. ASTM Designation: E8-61T. Pt. 30 of 1964 Book of ASTM Standards With Related Material. Amer. Soc. Testing Mater., c.1964, pp. 127-143.
9. Gray, Hugh R.: Hot-Salt Stress-Corrosion of Titanium Alloys: Generation of Hydrogen and Its Embrittling Effect. NASA TN D-5000, 1969.
10. Braski, David N.: Preliminary Investigation of Effect of Environmental Factors on Salt Stress Corrosion Cracking of Ti-8Al-1Mo-1V at Elevated Temperatures. NASA TM X-1048, 1964.
11. Braski, D. N.: Chemical Milling Solution Reveals Stress Corrosion Cracks in Titanium Alloy. NASA Tech Brief 67-10322, Sept. 1967.
12. Goldhoff, R. M.: Comparison of Parameter Methods for Extrapolating High-Temperature Data. Paper No. 58-A-121, Amer. Soc. Mech. Eng., 1958.



TABLE I

TENSILE PROPERTIES AND RESIDUAL TENSILE PROPERTIES  
OF Ti-6Al-4V TITANIUM-ALLOY SHEET  
[Exposure stress, 50 ksi (340 MN/m<sup>2</sup>)]

(a) Longitudinal properties of salt-coated tensile specimens

Exposure time, hr	Temperature		Yield strength		Tensile strength		Uniform elongation, percent
	°F	K	ksi	MN/m <sup>2</sup>	ksi	MN/m <sup>2</sup>	
400	650	620	(a)		81.6	563	0
400			(a)		73.7	508	0
150			129.2	891	140.4	968	5
150			(a)		119.8	826	0
100			118.2	815	119.2	822	0
100			132.8	916	142.0	979	5
50			128.9	889	138.7	956	4
50			130.0	896	142.0	979	7
1600			(a)		125.8	867	0
1600			(a)		129.7	894	0
1200	600	590	(a)		114.3	789	0
1200			128.7	887	130.3	898	0
800			(a)		130.8	902	0
800			(a)		129.9	896	0
400			131.2	905	132.8	916	4
400			136.2	939	138.9	958	4
1600			(a)		107.8	743	0
1600			126.5	872	129.0	889	1
1200			128.8	888	134.8	929	1
1200			(a)		128.3	885	0
800	550	560	137.2	946	142.8	985	3
800			137.3	947	145.3	1002	5

<sup>a</sup>Specimen fractured before yield stress was reached.

TABLE I.- Continued

TENSILE PROPERTIES AND RESIDUAL TENSILE PROPERTIES  
OF Ti-6Al-4V TITANIUM-ALLOY SHEET  
[Exposure stress, 50 ksi (340 MN/m<sup>2</sup>)]

(b) Transverse properties of salt-coated tensile specimens

Exposure time, hr	Temperature		Yield strength		Tensile strength		Uniform elongation, percent
	°F	K	ksi	MN/m <sup>2</sup>	ksi	MN/m <sup>2</sup>	
400	650	620	(a)		74.8	516	0
150			(a)		118.6	818	0
150			123.0	848	135.2	932	2
100			128.0	883	136.2	939	5
100			125.0	862	133.0	917	2
50			124.3	857	138.0	952	8
50			125.0	862	137.7	949	6
1600			(a)		110.0	758	0
1600	600	590	118.0	814	120.5	831	0
1200			125.6	866	126.6	873	1
1200			(a)		122.8	847	0
800			(a)		120.8	833	0
800			(a)		117.2	808	0
400			128.1	883	135.5	934	3
400			128.8	888	137.5	948	5
1600		550	128.6	887	133.8	923	3
1600			127.7	880	134.3	926	3
1200			128.1	883	136.0	938	5
1200			127.2	877	135.2	932	3
800			127.7	880	135.6	935	4
800			128.7	887	136.5	941	5

<sup>a</sup>Specimen fractured before yield stress was reached.

TABLE I. - Concluded

TENSILE PROPERTIES AND RESIDUAL TENSILE PROPERTIES  
OF Ti-6Al-4V TITANIUM-ALLOY SHEET  
[Exposure stress, 50 ksi (340 MN/m<sup>2</sup>)]

(c) Longitudinal and transverse properties of control tensile specimens

Exposure time, hr	Temperature		Yield strength		Tensile strength		Uniform elongation, percent
	°F	K	ksi	MN/m <sup>2</sup>	ksi	MN/m <sup>2</sup>	
Longitudinal							
1600	600	590	133.7	922	145.0	1000	9
Transverse							
1600	600	590	126.0	869	138.7	956	10
1600			126.0	869	138.2	953	8

(d) Uncoated, unexposed tensile specimens

Yield strength		Tensile strength		Young's modulus		Elongation, percent	
ksi	MN/m <sup>2</sup>	ksi	MN/m <sup>2</sup>	ksi	GN/m <sup>2</sup>	Uniform	2 in. (5 cm)
Longitudinal							
133.8	923	142.8	985	17 400.0	120.0	11	15
133.0	917	145.5	1003	17 700.0	122.0	11	14
Transverse							
129.9	896	137.8	950	16 400.0	113.8	10	14
126.8	874	136.3	938	16 000.0	110.3	11	15

TABLE II

STRESS-RUPTURE PROPERTIES OF SALT-COATED SPECIMENS  
OF Ti-6Al-4V TITANIUM-ALLOY SHEET

Temperature		Stress		Time to rupture, hr
°F	K	ksi	MN/m <sup>2</sup>	
700	650	80	552	128
		75	517	147
		70	483	214
		65	448	221
		60	414	380
		50	345	488
650	620	90	621	118
		85	586	158
		80	552	275
		75	517	426
		70	483	476
600	590	100	690	193
		95	655	336
		90	621	538
		85	586	578
		80	552	642

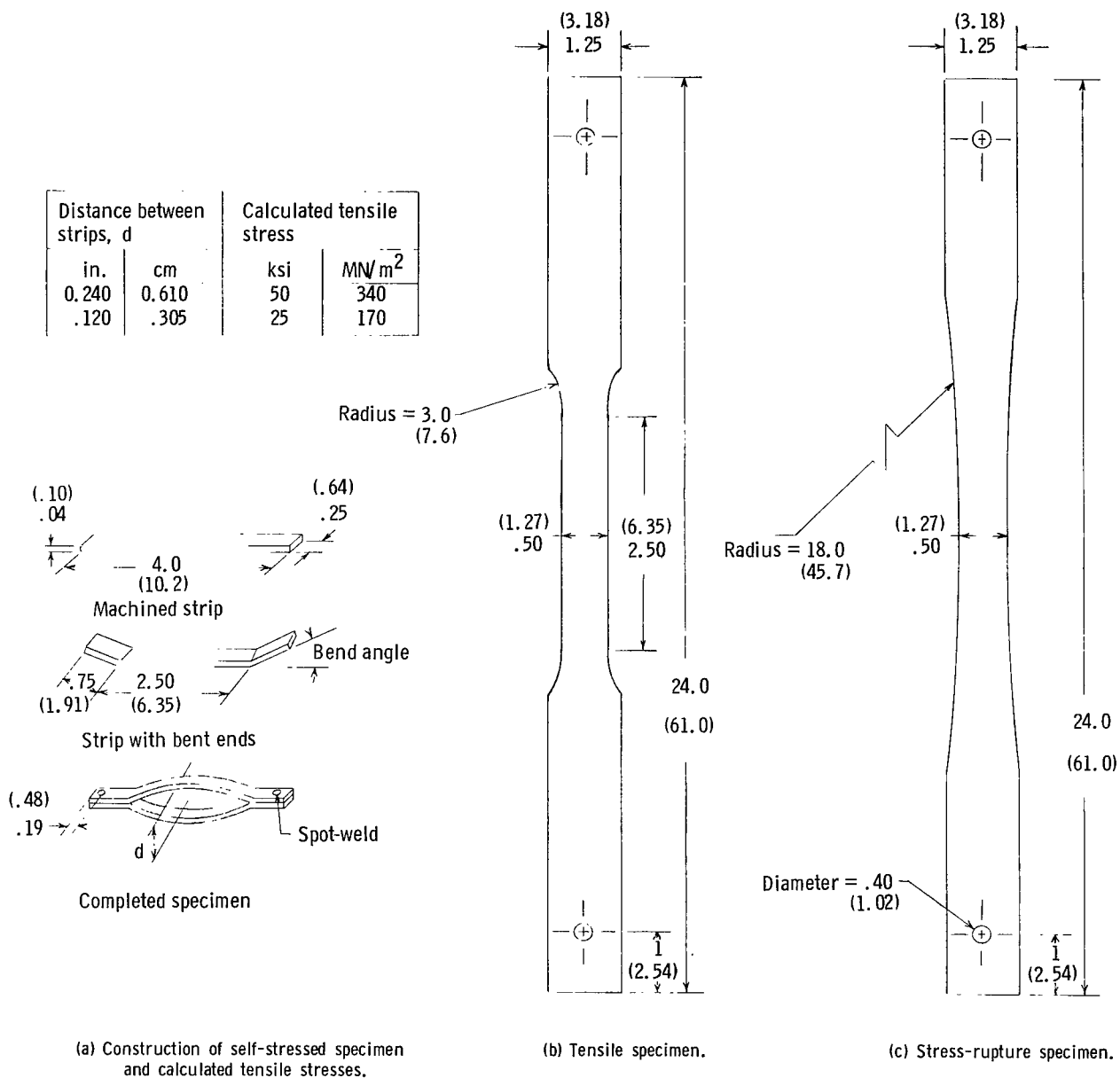
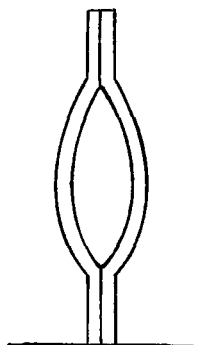


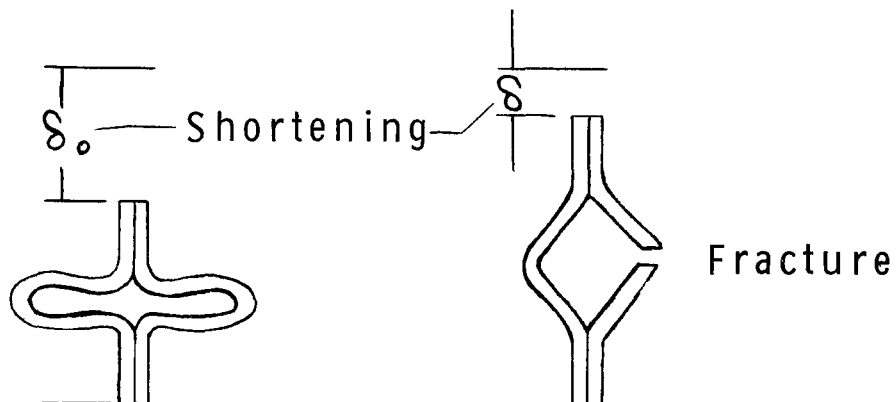
Figure 1.- Self-stressed, tensile, and stress-rupture specimens. Dimensions are in inches (centimeters).

Before test



Original  
specimen

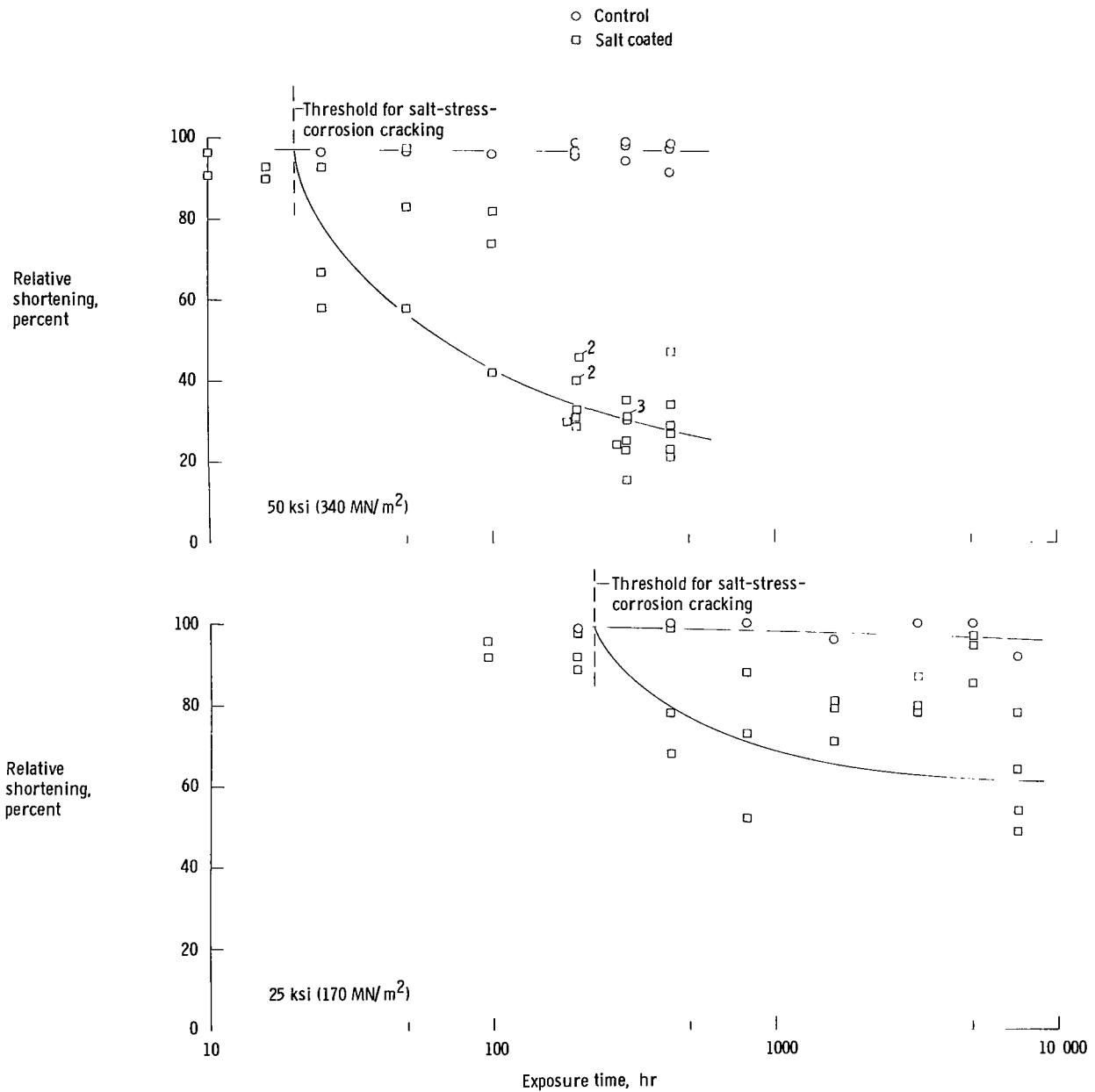
After test



Uncoated,  
unexposed

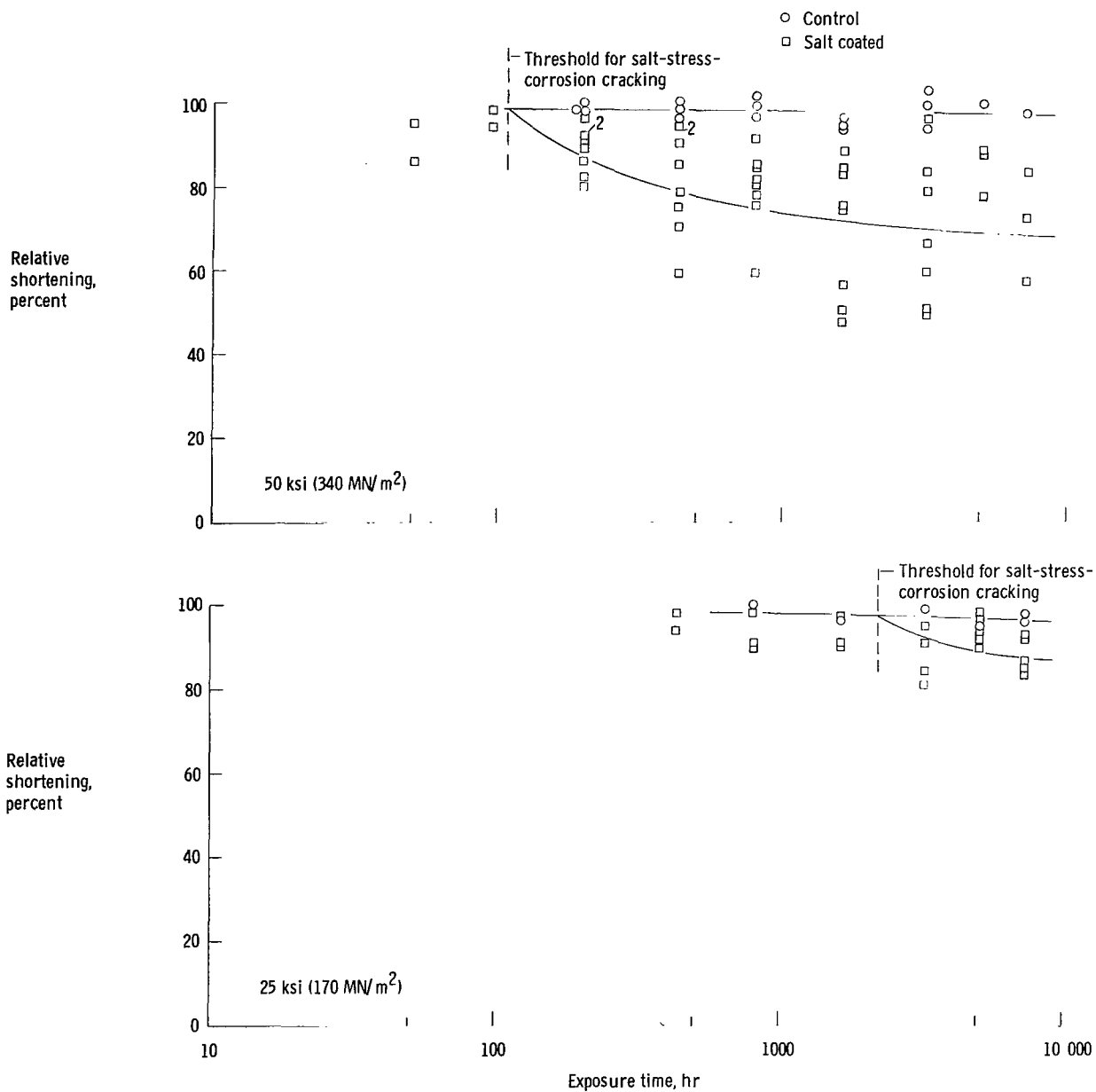
Salt-coated  
or control,  
exposed

Figure 2.- Specimen configuration before and after room-temperature compression test.



(a) 600° F (590 K).

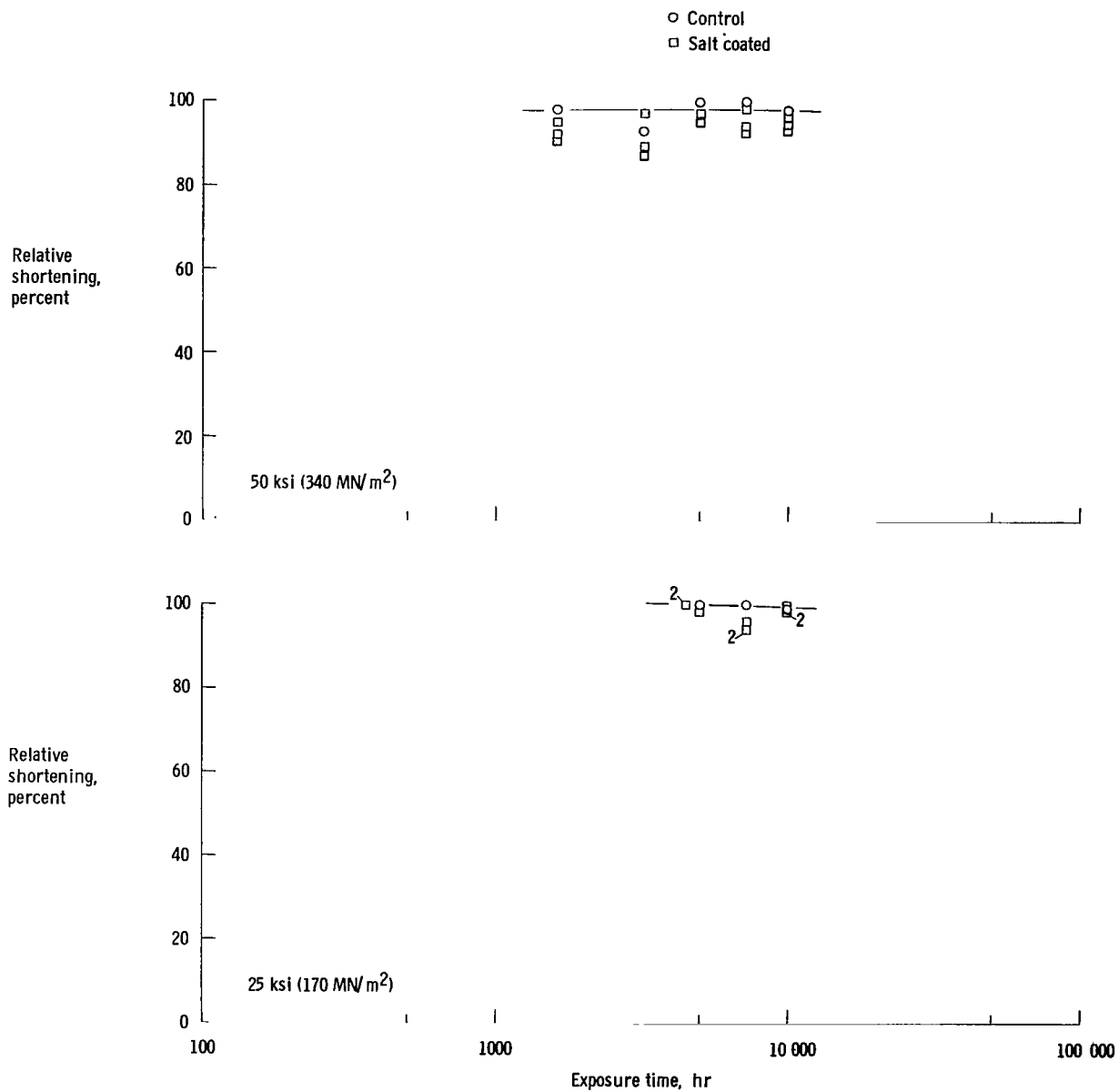
Figure 3.- Effect of various temperatures on hot-salt-stress corrosion of self-stressed specimens of annealed Ti-6Al-4V alloy sheet for 50 and 25 ksi (340 and 170 MN/m<sup>2</sup>) stress. (Numbers adjacent to test points indicate duplicate data.)



(b) 550° F (560 K).

Figure 3.- Continued.





(c) 500° F (530 K).

Figure 3.- Concluded.

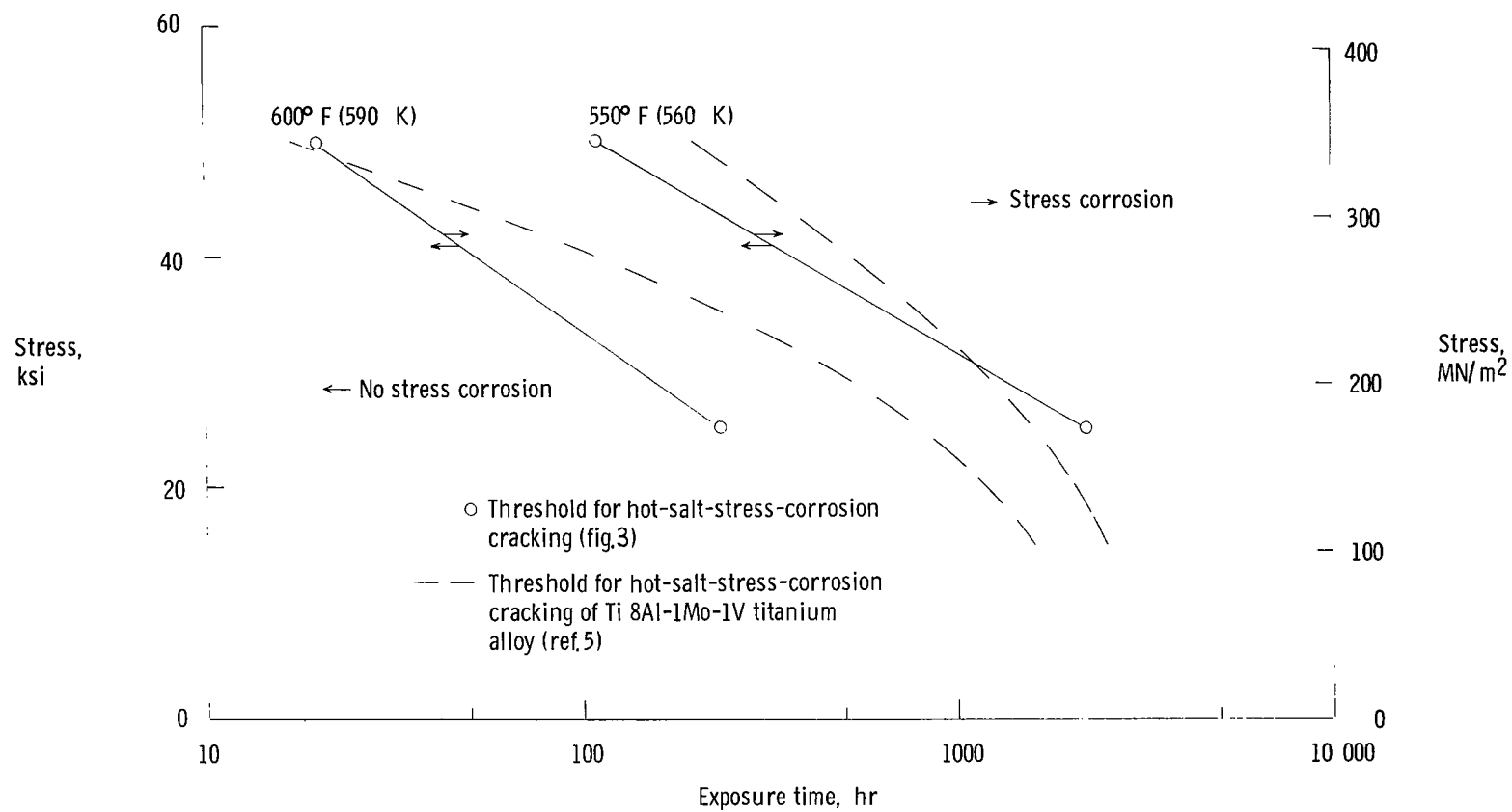


Figure 4.- Hot-salt-stress-corrosion crack threshold curves for Ti-6Al-4V and Ti-8Al-1Mo-1V titanium-alloy sheets exposed up to 10 000 hours.

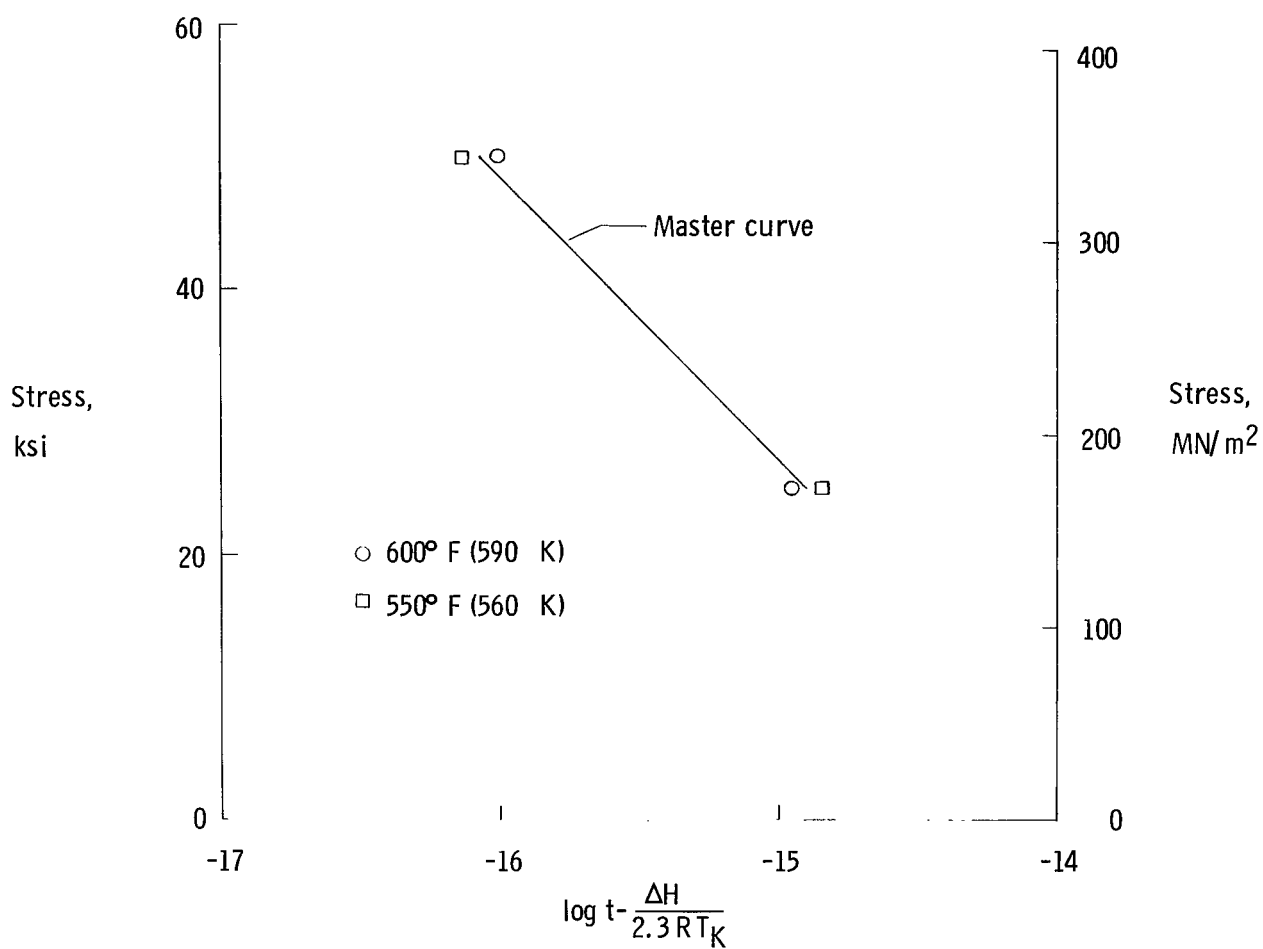
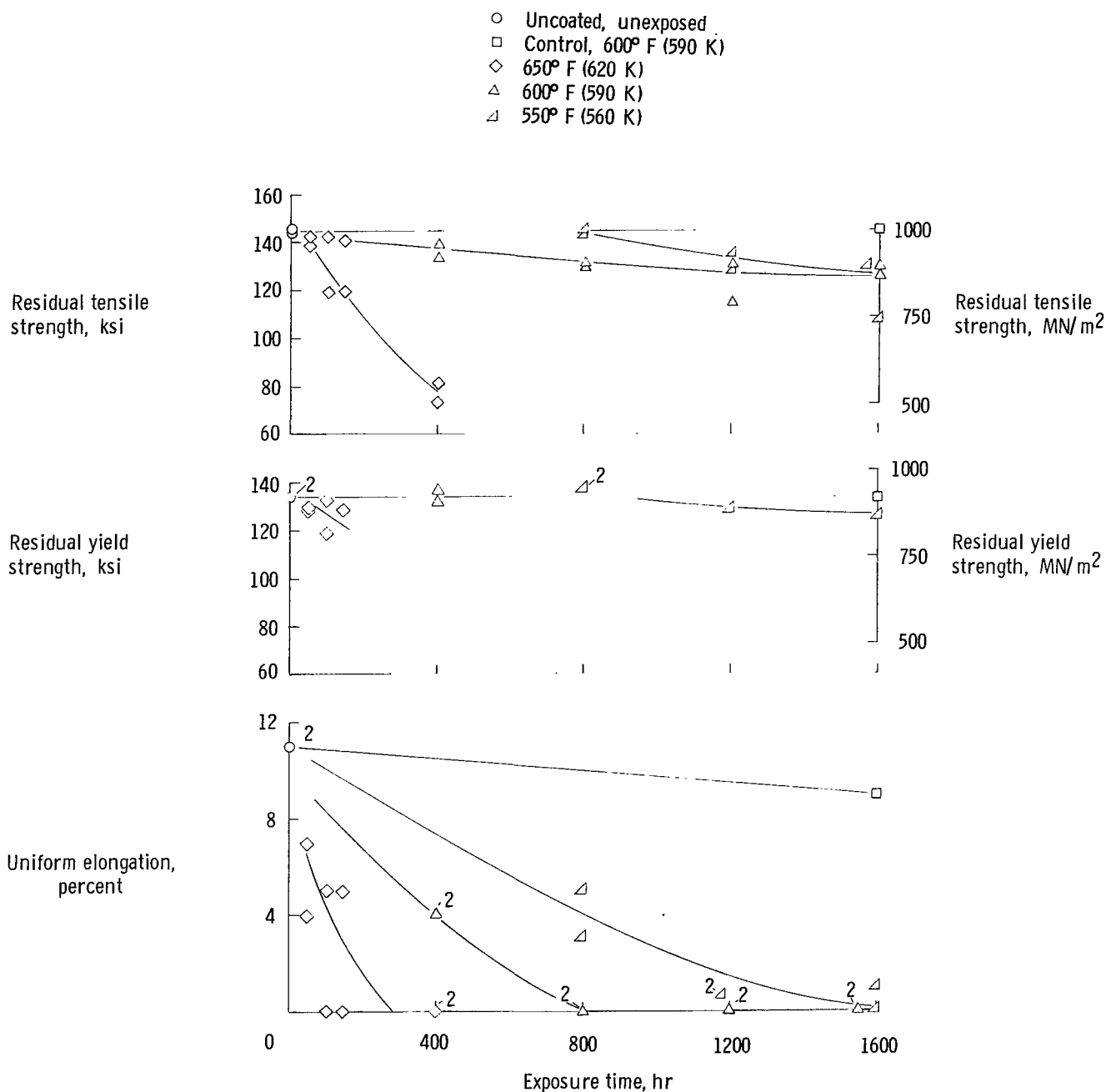
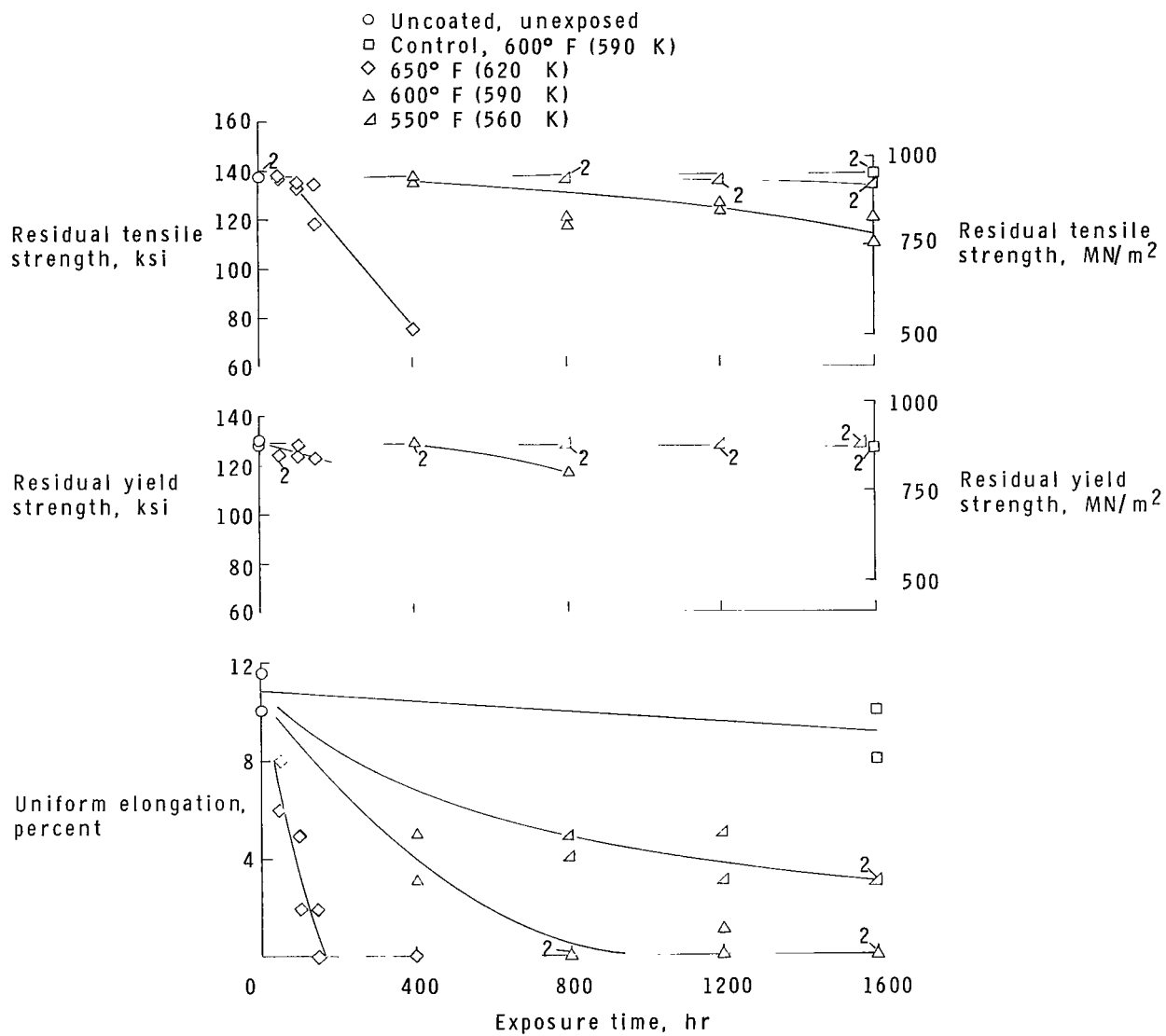


Figure 5.- Application of Orr-Sherby-Dorn parameter for predicting hot-salt-stress-corrosion crack initiation in Ti-6Al-4V titanium-alloy sheet.  
 $\Delta H = 47\,000$  cal/mole (197.0 kJ/mole).



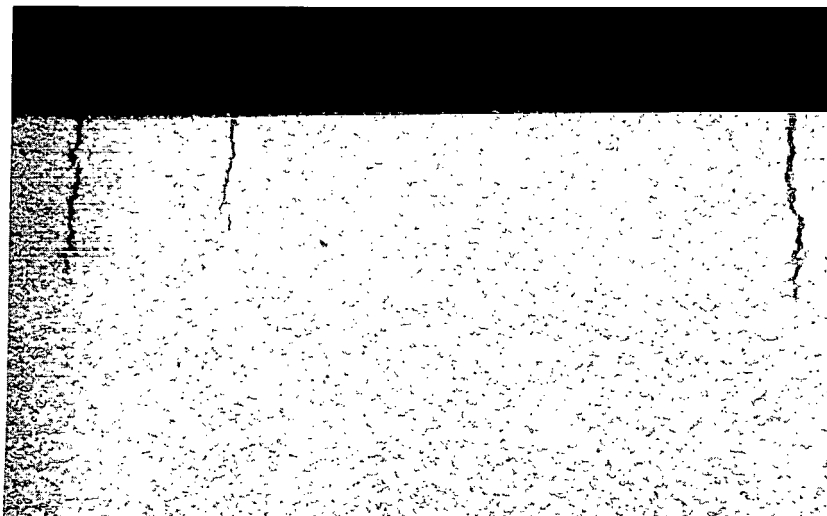
(a) Longitudinal specimens.

Figure 6.- Residual tensile properties for hot-salt-stress-corrosion-cracked Ti-6Al-4V titanium-alloy sheet after exposures up to 1600 hours at 50 ksi (340 MN/m<sup>2</sup>) stress from 550° F to 650° F (560 K to 620 K).



(b) Transverse specimens.

Figure 6.- Concluded.



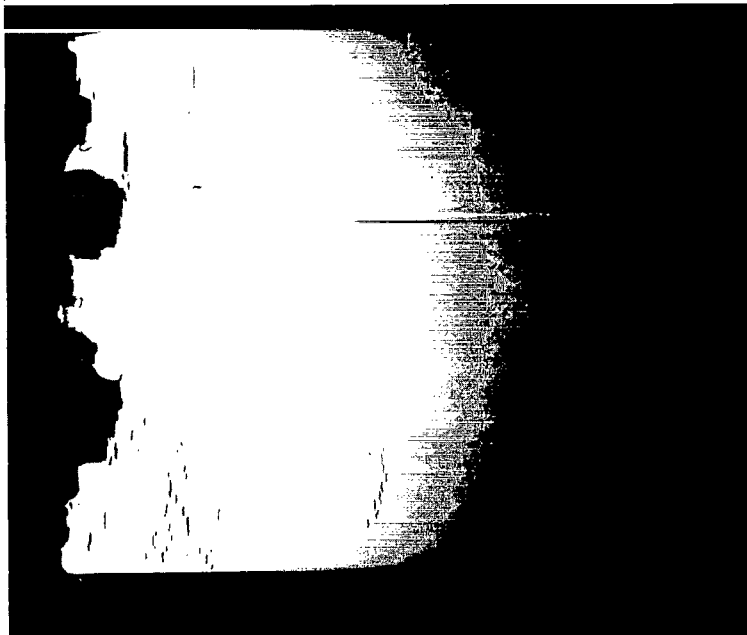
(a) Magnification, 100×.



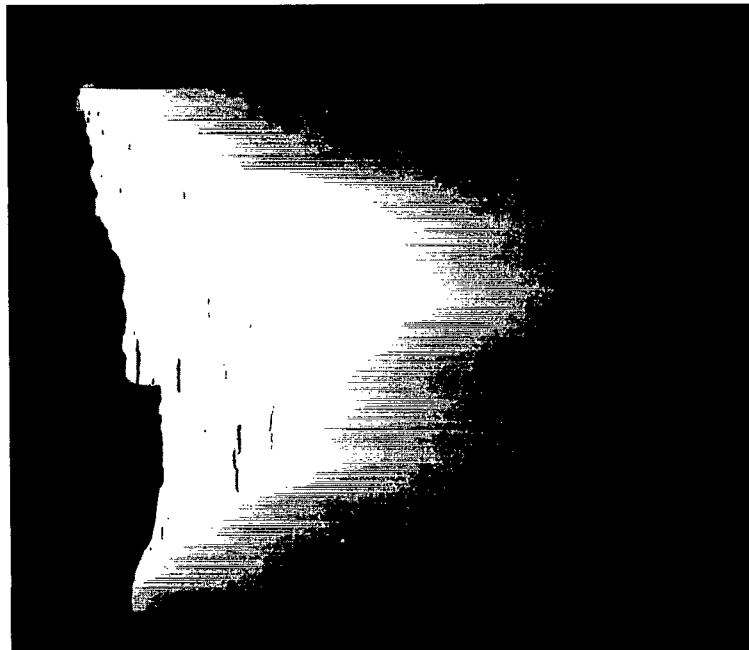
(b) Magnification, 500×.

Figure 7.- Edge view of hot-salt-stress-corrosion cracks in longitudinal tensile specimen of Ti-6Al-4V titanium-alloy sheet exposed at 50 ksi (340 MN/m<sup>2</sup>) and 600° F (590 K) for 800 hours.

L-69-5251



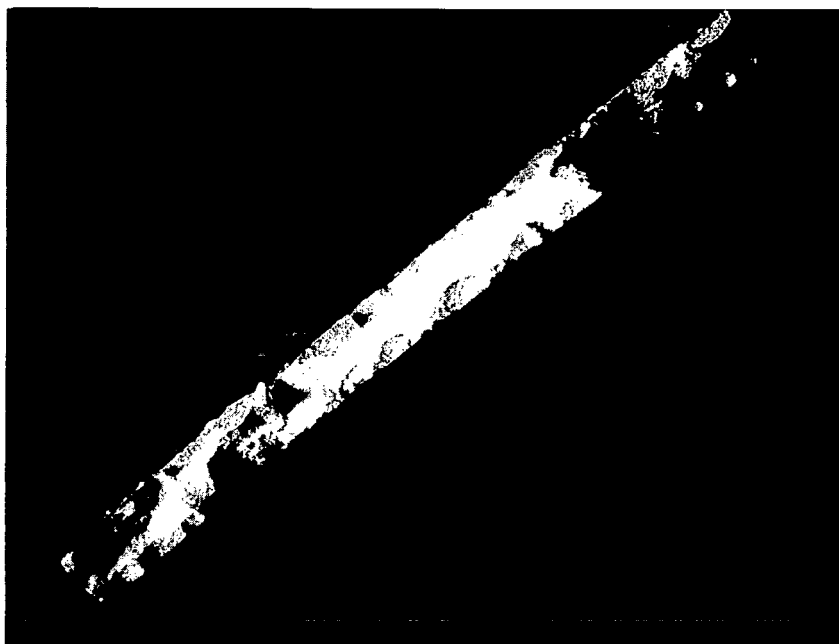
Longitudinal



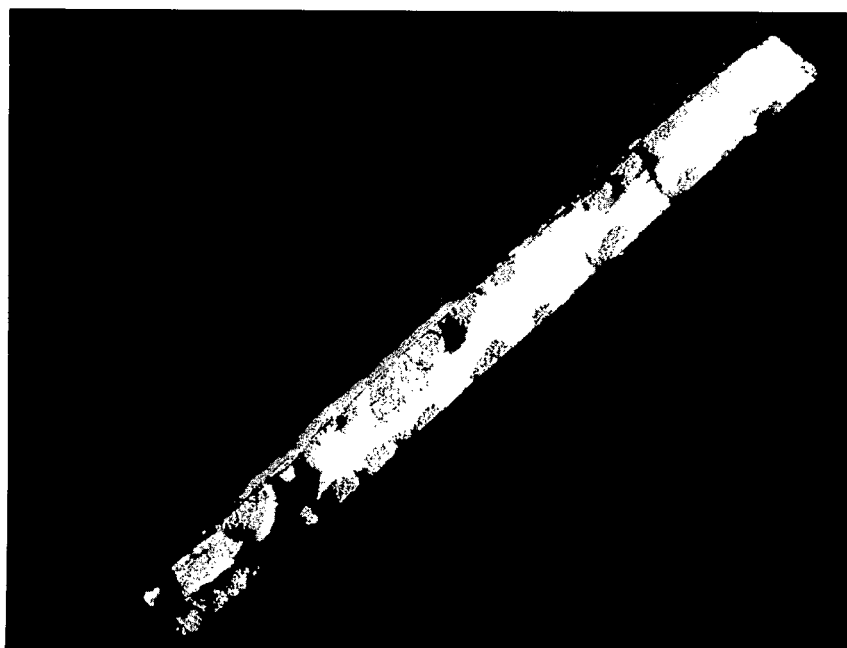
Transverse

Figure 8.- Hot-salt-stress-corrosion surface cracks in tensile specimens of Ti-6Al-4V titanium-alloy sheet after 1600 hours exposure at 50 ksi (340 MN/m<sup>2</sup>) stress. Fracture surface to the left of each photograph. Magnification, 6×.

L-69-5252



Longitudinal



Transverse

L-69-5253

Figure 9.- Fracture surfaces of tensile specimens of Ti-6Al-4V titanium alloy revealing hot-salt-stress-corrosion cracks along the perimeter of the fracture. Magnification, 9 $\times$ .



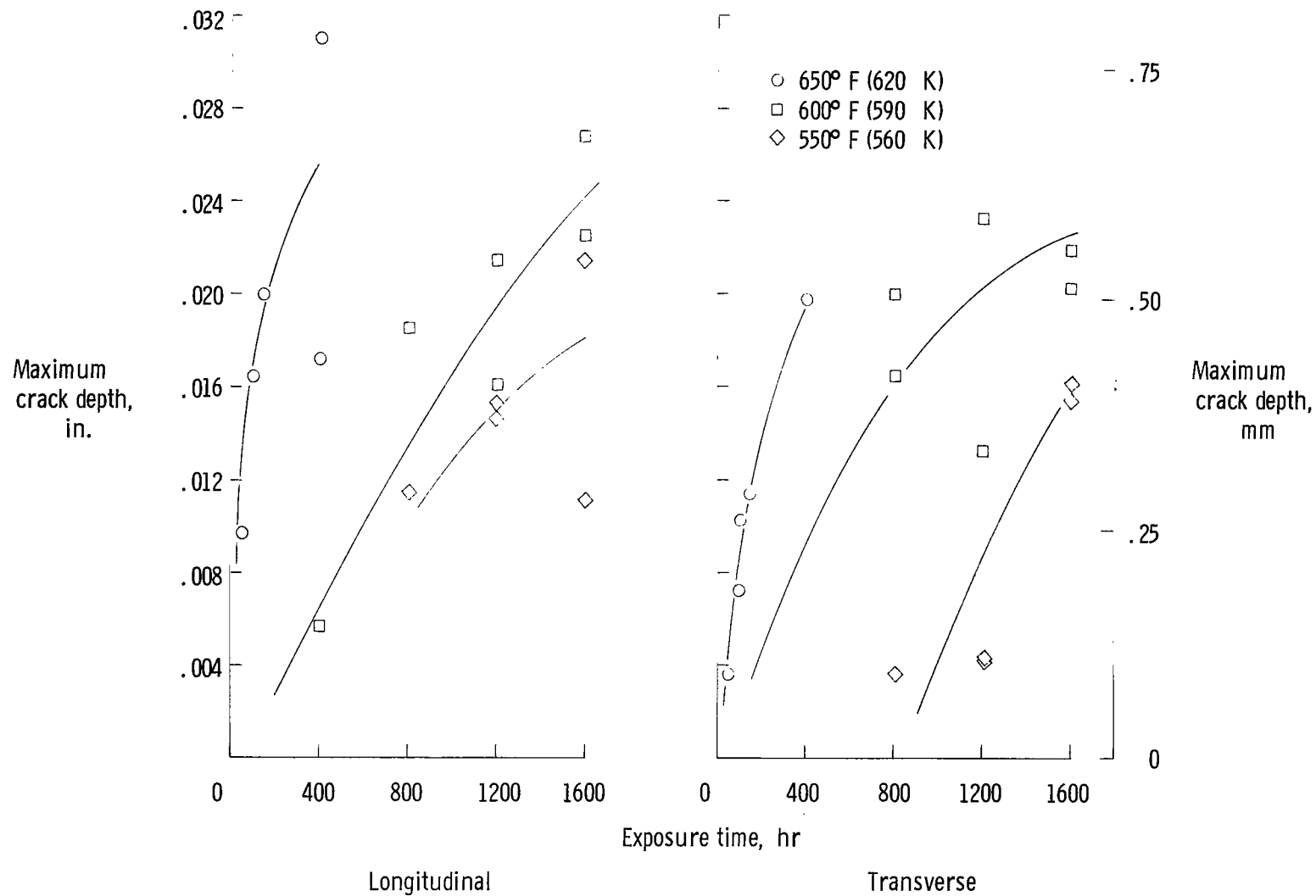


Figure 10.- Hot-salt-stress-corrosion maximum crack penetration for tensile specimens of Ti-6Al-4V titanium-alloy sheet for exposures up to 1600 hours at 50 ksi (340 MN/m<sup>2</sup>) stress.

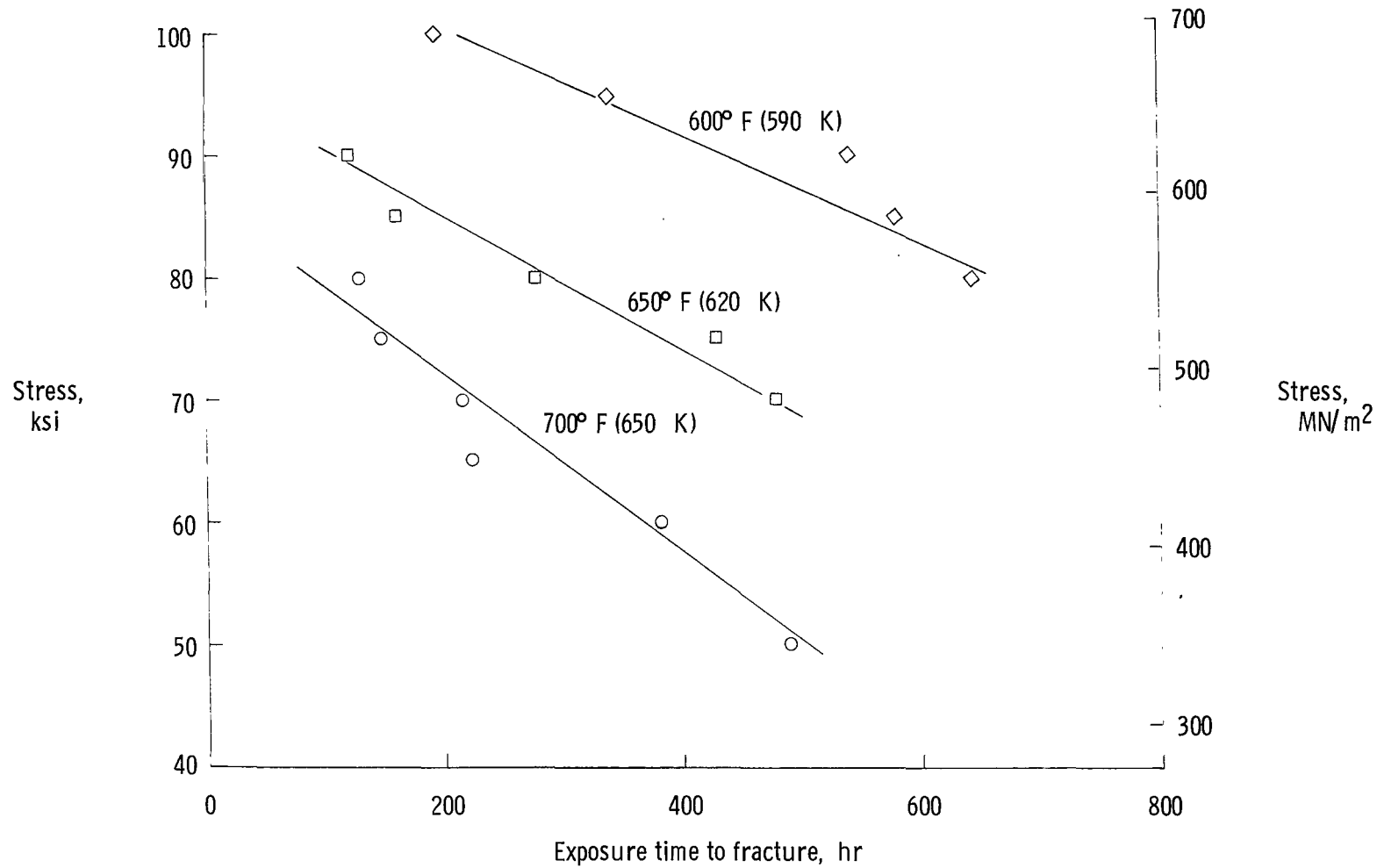


Figure 11.- Stress-rupture properties for salt-coated Ti-6Al-4V titanium-alloy sheet for exposures from 600° F to 700° F (590 K to 650 K) and stresses up to 100 ksi (680  $\text{MN/m}^2$ ).



L-69-5254  
Figure 12.- Fracture surface of stress-rupture specimen of Ti-6Al-4V titanium alloy revealing the picture frame effect produced by the hot-salt-stress-corrosion cracks along the perimeter of the fracture. Magnification, 9 $\times$ .

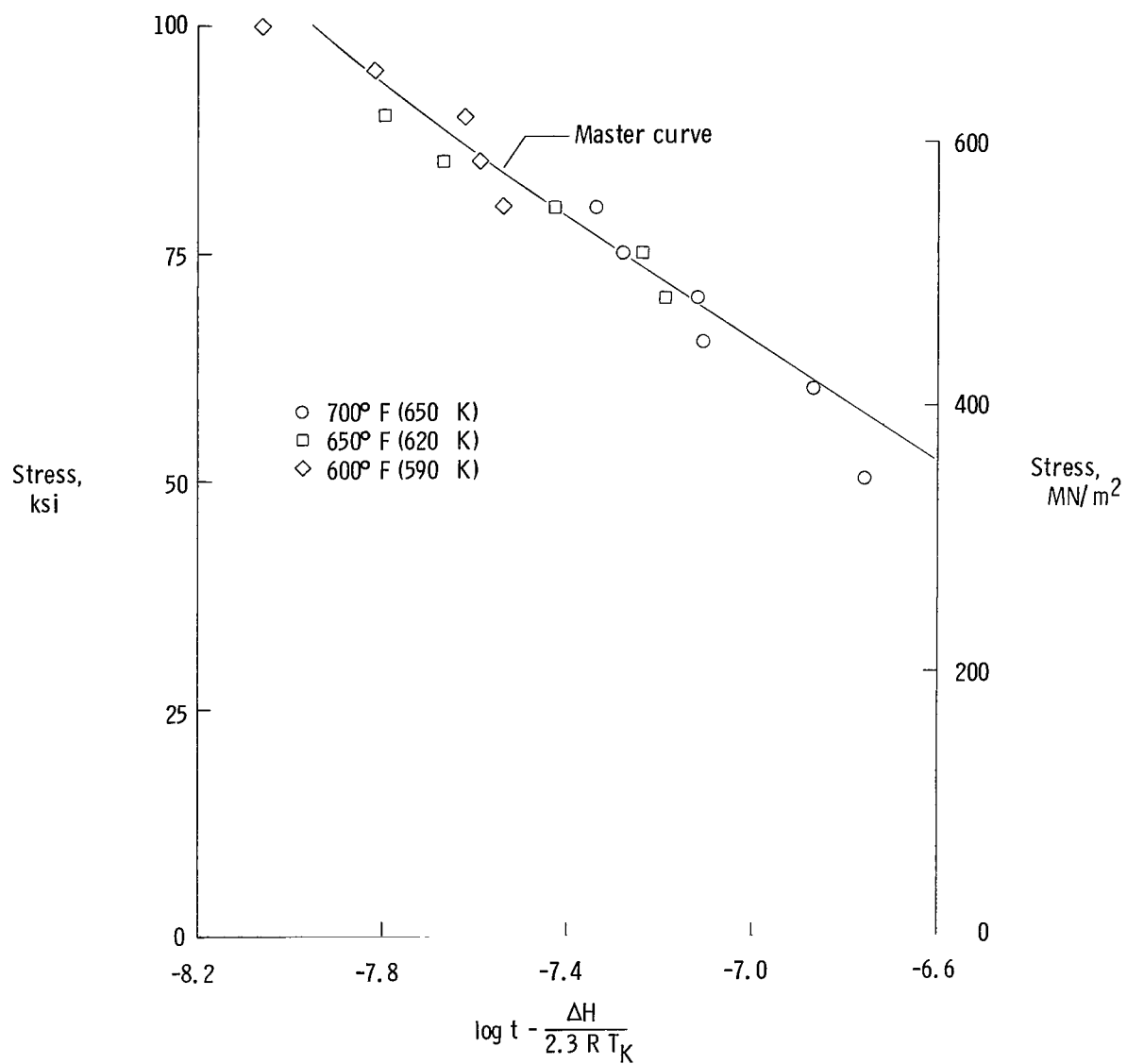


Figure 13.- Application of Orr-Sherby-Dorn parameter for predicting stress rupture of salt-coated specimens in Ti-6Al-4V titanium-alloy sheet.  
 $\Delta H = 28\,000 \text{ cal/mole}$  ( $117.0 \text{ kJ/mole}$ ).

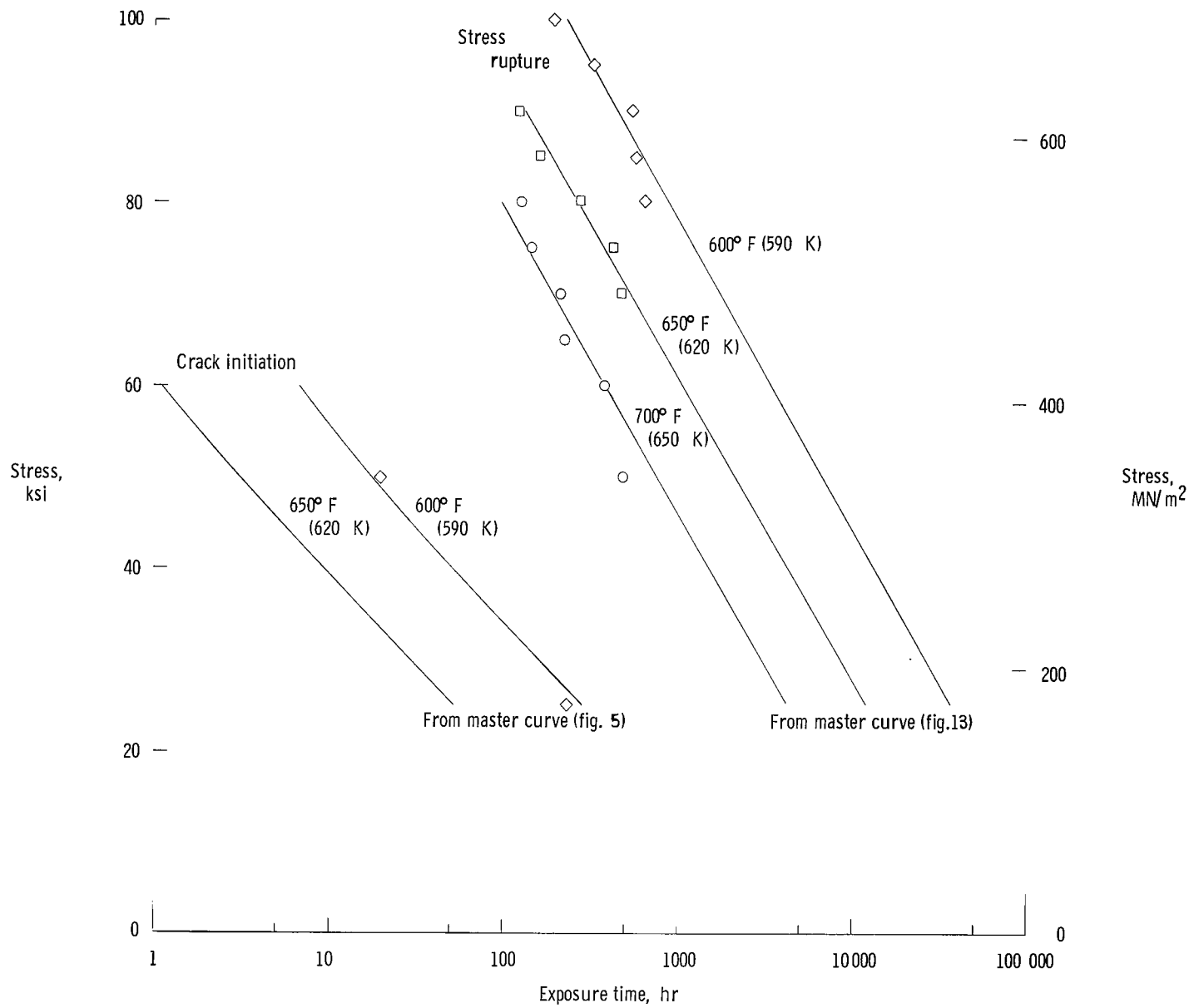


Figure 14.- Relation of hot-salt-stress-corrosion crack initiation to stress rupture of salt-coated specimens of Ti-6Al-4V titanium-alloy sheet.

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